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DETAILED DESCRIPTION

[Detailed Description of the Invention]

Field of the invention on industry This invention relates to the outstanding electrical conductivity and thermal conductivity which compress a thermal expansion vapor growth graphite fiber, and change, a heat-resistant and chemical-resistant flexible graphite sheet material, and its manufacture approach.

The conventional technique It is immersed, a graphite is made to react conventionally into mixed liquor with a fuming nitric acid or concentrated sulfuric acid and concentrated nitric acid, a nitrate, perchloric acid, a chlorate, a chromic acid, a chromate, or dichromic acid, a lot of water is added and washed to the generated graphite lamellar compound, and the manufacture approach of the thermal expansion nature graphite which consists of subsequently drying at the temperature of 100 degrees C or more is learned. Furthermore, the manufacture approach of the thermal expansion nature graphite which consists of processing the graphite powder of the natural graphite which distributed and slurred, pyrolytic graphite, or kish graphite with the peroxomonosulfuric acid generated at the temperature of 15-60 degrees C according to an operation of the hydrogen peroxide of 30% or more of concentration and concentrated sulfuric acid in concentrated sulfuric acid is proposed (JP,60-34492,B).

Moreover, add the oxidizer of the mixture (concentration: 0.1N, 1.0N, or 10Ns) of the concentrated sulfuric acid of the graphite powder of a natural graphite, pyrolytic graphite, or kish graphite, and a nitric acid, and it processes in ordinary temperature. Subsequently, by generating the graphite which heated at 1000 degrees C and expanded in the direction of "C" of a original particle, after rinsing, and compressing into at least 80kg consistency of//m3 under un-existing [of adhesives] The method of obtaining the flexible graphite sheet material which has the tensile strength of 1.56 - 225 kg/cm2 at a room temperature by 0.00254-12.7mm in thickness is proposed (JP,44-23966,B).

By the above-mentioned well-known approach, graphite powder, such as a natural graphite, pyrolytic graphite, and kish graphite, is used as a raw material. however, the flexible graphite sheet material which used as the raw material the expanded graphite obtained from these graphite powder -- the hexagon-head tabular of the hexagonal system of a graphite -- it laps with "C" shaft orientations in the shape of a layer as a flat thin crystal. Therefore, the property in which electrical conductivity, thermal conductivity, a dynamics property, etc. were excellent only about "a" and "b" shaft orientations speaks (it is the thickness direction where c shaft orientations are vertical to a sheet surface, and is the direction where a and b shaft orientations are parallel to a sheet surface.).

For example, the flexible graphite sheet material currently indicated by JP,44-23966,B has a remarkable low fault as the electric specific resistance and the thermal conductivity of the thickness direction (c shaft orientations) are the following as compared with the direction of a field (a and b shaft orientations), respectively.

| | 面方向 (a及びb軸方向) | 厚み方向 (c軸方向) |
|-------------------------------|------------------|----------------|
| 電気比抵抗($\mu\Omega \cdot cm$) | 700 | 30,000 |
| 熱伝導度 (Kcal/m・hr・°C) | 120 | 4 |

Therefore, it is anxious for offer of the flexible graphite sheet material which has the outstanding electrical conductivity and thermal conductivity with the electric specific resistance and the thermal conductivity of c shaft orientations, i.e., the thickness direction, almost equivalent to a and b shaft orientations of a field, i.e., the direction.

The result of having inquired wholeheartedly so that this invention person etc. may get a flexible graphite sheet material with the electric specific resistance and the thermal conductivity of c shaft orientations almost

equivalent to the electric specific resistance of a and b shaft orientations, and thermal conductivity, The vapor growth graphite fiber which has the nearly perfect graphite structure near the natural graphite obtained by carrying out graphitization processing of the vapor growth carbon fiber which has annual-rings structure is processed with an oxidizing quality concentrated-sulfuric-acid solution. The mixture of the obtained thermal expansion nature vapor growth graphite fiber or this thermal expansion nature vapor growth graphite fiber, and thermal expansion nature graphite powder is heated and expanded in temperature of 400-1700 degrees C. Subsequently, based on a header and this knowledge, it came to accomplish this invention for the obtained graphite sheet having the electrical conductivity and thermal conductivity which were excellent in all the directions of a, b, and c axis by compressing.

Configuration of invention The vapor growth graphite fiber obtained as a vapor growth graphite fiber of this invention by the approach currently indicated by JP,41-12091,B, JP,50-64527,A, JP,52-103528,A, JP,58-180615,A, JP,60-27700,A, and Japanese Patent Application No. 61-92507 can be illustrated.

For example, it is made to circulate within [which is held with the carrier gas of inert gas, such as hydrogen or nitrogen gas, in hydrocarbon gas, such as benzene, toluene, methane and ethane, at 1000-1300 degrees C] a reaction. And/or, a carbon fiber (carbon whisker) is grown up by using the metal particle of iron or transition metals as a catalyst nucleus. the product made from graphite within [this] a reaction, or the nucleation band on the base made from porcelain -- Subsequently, a vapor growth graphite fiber can be obtained by carrying out graphitization processing of the obtained carbon fiber 1 minute or more at the temperature of 2400 degrees C or more.

** -- unlike the graphite fiber manufactured from the PAN system or the pitch based carbon fiber, the vapor growth graphite fiber obtained by making it like has nearly perfect annual-rings-like graphite structure. That is, the thermal conductivity is as follows at the electrical conductivity list of the fiber shaft orientations of the vapor growth graphite fiber used by this invention, and the growth direction (direction which goes to a fiber axis direct) of annual rings.

(纖維軸方向) (年輪の成長方向)

| | | |
|---------------------------------------|---------|-------|
| ・電気比抵抗 ($\mu\Omega \cdot cm$) | 60~100 | 10000 |
| ・熱伝導度 (Kcal/m ² ・hr・°C) | 600~900 | — |

The vapor growth graphite fiber which has these properties A fuming nitric acid, or concentrated sulfuric acid and a nitric acid, Potassium chlorate, a chromic acid, potassium permanganate, perchloric acid, a hydrogen peroxide, With an oxidizing quality concentrated-sulfuric-acid mixed solution with the oxidizer of a sulfuric anhydride or persulfuric acid ammonium, process for 10 - 90 minutes, add and rinse 15-60 degrees C of a lot of water to the acquired resultant between graphite layers, and it dries at the temperature of 90 degrees C or more.

Subsequently, a thermal expansion vapor growth graphite fiber can be obtained by heat-treating at 400-1700 degrees C. The obtained thermal expansion vapor growth graphite fiber shows the same property as the thermal expansion graphite obtained from the natural graphite.

The above-mentioned reaction between graphite layers can illustrate the approach of adding a vapor growth graphite fiber gradually and agitating it more than for 30 minutes, while it is desirable to carry out by processing with the mixed solution of concentrated sulfuric acid and a hydrogen peroxide, for example, it keeps the temperature of the mixed solution of a vapor growth graphite fiber which was mixed 40 or less % of the weight so that it might become 5 - 20 % of the weight preferably, and was subsequently obtained at 20-50 degrees C for concentrated sulfuric acid of 90% or more of concentration, and hydrogen peroxide solution of 30 % of the weight or more of concentration.

Mixture of the thermal expansion nature vapor growth graphite fiber independent or this thermal expansion nature vapor growth graphite fiber obtained as mentioned above, and an expansibility graphite (a mixed rate 50 or less % of the weight of vapor growth graphite fibers) Preferably, at 400-1700 degrees C, heat-treat 10 - 30 % of the weight, and it is expanded. Subsequently, a flexible graphite sheet material is preferably obtained at least 0.9 or more under existence of adhesives or nonexistence the thickness of 0.1-10mm, and by compressing into the umbrella consistency of 1.0-1.5 under a room temperature.

Since the thermal expansion vapor growth graphite fiber is entangled, and since a thermal expansion graphite exists between the thermal expansion vapor growth graphite fibers which became entangled, the electrical conductivity of the thickness direction and thermal conductivity become high in ****, and the obtained flexible graphite sheet material has, outstanding sheet reinforcement, for example, tensile strength.

Effect of the invention The flexible graphite sheet material of this invention Thermal conductivity below by 500micro ohm-cm preferably below 700micro ohm-cm More than 120 Kcal/m-hr and ** [the electric specific

resistance of the direction of a field of a sheet] More than 150 Kcal/m-hr and ** are shown preferably. The electric specific resistance of the thickness direction of a sheet Below 10,000micro ohm-cm Thermal conductivity below by 5,000micro ohm-cm preferably More than 10 Kcal/m-hr and ** It is the flexible graphite sheet material in which more than 20 Kcal/m-hr and ** are shown preferably and which tensile strength shows the electrical conductivity, the outstanding thermal conductivity, and outstanding reinforcement which have 40 - 70 kg/cm² preferably two or more 40 kg/cm.

Furthermore, the flexible graphite sheet material of this invention also shows the outstanding thermal resistance and chemical resistance.

Example - 1 and 2 Having added 60% hydrogen peroxide solution of the 20 sections, carrying out cooling churning of the sulfuric-acid 400 section 98%, and adding the vapor growth graphite fiber 100 section small quantity every in a 20-50-degree C temperature requirement subsequently, churning was continued for 60 minutes and the reaction between layers was performed. After the reaction, the product dried with the 100**5-degree C oven, after washing with sufficient quantity of water.

The mixture (a mixed rate is 10:90) (example -2) of independent (example -1) or this thermal expansion nature vapor growth graphite fiber, and a thermal expansion nature graphite is heat-treated with a 1000-degree C electric furnace, respectively, the obtained thermal expansion nature vapor growth graphite fiber was expanded, subsequently it compressed under the nonexistence of adhesives, and the flexible graphite sheet of the thickness of 0.5mm and the umbrella consistency 1.0 was obtained. The electrical conductivity, the thermal conductivity, and tensile strength of the sheet are as follows.

表

| | 抗張力 (kg/cm ²) | 電気比抵抗 ($\mu\Omega \cdot \text{cm}$) | | 熱伝導度 (Kcal/m ² · hr · °C) | |
|-------|------------------------------|--|-------|---|------|
| | | 面方向 | 厚み方向 | 面方向 | 厚み方向 |
| 実施例-1 | 70 | 60 | 100 | 600 | 400 |
| 実施例-2 | 42 | 550 | 4,200 | 140 | 25 |

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CLAIMS

(57) [Claim(s)]

[Claim 1] the electric specific resistance of the direction of a field of a sheet -- the flexible graphite sheet material which tensile strength is [thermal conductivity / thermal conductivity] two or more 40 kg/cm more than 10 Kcal/m-hr and ** more than 120 Kcal/m-hr and ** for the electric specific resistance of the thickness direction of a sheet, and an umbrella consistency is 0.9 or more, and consists of a thermal expansion vapor growth graphite fiber at least below by 10,000micro ohm-cm by below 700micro ohm-cm.

[Claim 2] A vapor growth graphite fiber A fuming nitric acid or concentrated sulfuric acid and a nitric acid, potassium chlorate, a chromic acid, Process with an oxidizing quality concentrated-sulfuric-acid mixed solution with potassium permanganate, perchloric acid, a hydrogen peroxide, a sulfuric anhydride, or persulfuric acid ammonium, and a thermal expansion nature vapor growth graphite fiber is obtained. The manufacture approach of the flexible graphite sheet material which consists of obtaining the obtained thermal expansion vapor growth graphite fiber independent or the graphite which heat-treated mixture with a thermal expansion nature graphite at 400-1700 degrees C, and expanded, and compressing the obtained graphite into an umbrella consistency and 0.9 or more under existence of adhesives or nonexistence.

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(54) 【発明の名称】 可撓性黒鉛シート材料

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(57) 【特許請求の範囲】

【請求項 1】 シートの面方向の電気比抵抗が $700 \mu \Omega \cdot \text{cm}$ 以下で熱伝導度が $120 \text{Kcal/m} \cdot \text{hr} \cdot ^\circ\text{C}$ 以上、シートの厚み方向の電気比抵抗が $10,000 \mu \Omega \cdot \text{cm}$ 以下で熱伝導度が $10 \text{Kcal/m} \cdot \text{hr} \cdot ^\circ\text{C}$ 以上、抗張力が 40Kg/cm^2 以上で、且つかサ密度が 0.9 以上であり、少なくとも熱膨張気相成長黒鉛繊維から成る可撓性黒鉛シート材料。

【請求項 2】 気相成長黒鉛繊維を発煙硝酸又は濃硫酸と硝酸、塩素酸カリウム、クロム酸、過マンガン酸カリウム、過塩素酸、過酸化水素、無水硫酸又はベルオキソ硫酸アンモニウムとの酸化性濃硫酸混合溶液で処理して熱膨張性気相成長黒鉛繊維を得、得られた熱膨張気相成長黒鉛繊維単独又は、熱膨張性黒鉛との混合物を $400 \sim 170^\circ\text{C}$ に加熱処理して膨張した黒鉛を得、得られた黒鉛を接着剤の存在下又は非存在下でかさ密度、0.9 以上に圧

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縮することから成る可撓性黒鉛シート材料の製造方法。

【発明の詳細な説明】

産業上の利用分野

本発明は熱膨張気相成長黒鉛繊維を圧縮して成る、優れた電気伝導性、熱伝導性、耐熱性及び耐薬品性の可撓性黒鉛シート材料及びその製造方法に関する。

従来技術

従来、発煙硝酸又は濃硫酸と濃硝酸、硝酸塩、過塩素酸、塩素酸塩、クロム酸、クロム酸塩又は重クロム酸との混合液中に黒鉛を浸漬して反応せしめ、生成した黒鉛層間化合物に多量の水を加えて洗浄し、次いで 100°C 以上の温度で乾燥することから成る熱膨張性黒鉛の製造方法が知られている。更に、濃硫酸中に分散、スラリー化した天然黒鉛、熱分解黒鉛又はキッシュ黒鉛の黒鉛粉末を $15 \sim 60^\circ\text{C}$ の温度で濃度 30% 以上の過酸化水素及び濃硫

酸の作用により生成したベルオキソー硫酸で処理することから成る熱膨張性黒鉛の製造方法が提案されている（特公昭60-34492）。

また、天然黒鉛、熱分解黒鉛又はキッシュ黒鉛の黒鉛粉末の濃硫酸と硝酸との混合物（濃度：0.1N、1.0N又は10N）の酸化剤を加えて常温で処理し、次いで水洗した後1000°Cに加熱して原子子の“c”方向に膨張された黒鉛を生成し、接着剤の不存在下で少なくとも80Kq/cm²の密度に圧縮することによって、厚さ0.00254～12.7mmで室温で1.56～225Kq/cm²の抗張力を有する可撓性黒鉛シート材料を得る方法が提案されている（特公昭44-23966）。

上述の公知の方法では、天然黒鉛、熱分解黒鉛、キッシュ黒鉛等の黒鉛粉末を原料として使用している。しかしながら、これら黒鉛粉末から得られた膨張黒鉛を原料とした可撓性黒鉛シート材料は、黒鉛の六方晶系の六角板状扁平な薄い結晶で“c”軸方向に層状に重なったものである。従って電気伝導性、熱伝導性及び力学特性等は“a”及び“b”軸方向についてのみ優れた特性が発言する（c軸方向とはシート面に垂直な厚み方向であり、a及びb軸方向とはシート面に平行な方向である。）。

例えば、特公昭44-23966に開示されている可撓性黒鉛シート材料はその面方向（a及びb軸方向）に比較して厚さ方向（c軸方向）の電気比抵抗及び熱伝導度がそれぞれ下記の通り著しく低い欠点がある。

| | 面方向 (a及びb軸方向) | 厚み方向 (c軸方向) |
|---|------------------|----------------|
| 電気比抵抗($\mu\Omega \cdot \text{cm}$) | 700 | 30,000 |
| 熱伝導度 ($\text{kcal}/\text{m} \cdot \text{hr} \cdot ^\circ\text{C}$) | 120 | 4 |

従って、c軸方向すなわち厚み方向の電気比抵抗及び熱伝導度がa及びb軸方向すなわち面方向とはほぼ同等である優れた電気伝導性及び熱伝導性を有する可撓性黒鉛シート材料の提供が切望されている。

本発明者等は、c軸方向の電気比抵抗及び熱伝導度がa及びb軸方向の電気比抵抗及び熱伝導度とはほぼ同等である可撓性黒鉛シート材料を得るべく鋭意研究をした結果、年輪構造を有する気相成長炭素繊維を黒鉛化処理することによって得られた天然黒鉛に近いほぼ完全な黒鉛構造を有する気相成長黒鉛繊維を酸化性濃硫酸溶液で処理し、得られた熱膨張性気相成長黒鉛繊維又は該熱膨張性気相成長黒鉛繊維と熱膨張性黒鉛粉末との混合物を400～1700°Cの温度に加熱して膨張させ、次いで圧縮することによって得られた黒鉛シートはa、b及びc軸のすべての方向において優れた電気伝導性及び熱伝導性を有することを見出し、この知見に基づいて本発明を成すに至った。

発明の構成

本発明の気相成長黒鉛繊維としては、特公昭41-1209

1、特開昭50-64527、特開昭52-103528、特開昭58-18061

5、特開昭60-27700及び特願昭61-92507に開示されている方法で得られた気相成長黒鉛繊維を例示し得る。

例えば、ベンゼン、トルエン、メタン、エタン等の炭化水素ガスを水素又は窒素ガス等の不活性ガスのキャリアガスと共に1000～1300°Cに保持されている反応管内を流通させて、該反応管内のグラファイト製又は磁器製基盤上の核生成帯域に、及び／又は鉄あるいは遷移金属の金属微粒子を触媒核として炭素繊維（炭素ウィスカー）を成長させ、次いで得られた炭素繊維を2400°C以上の温度で1分以上黒鉛化処理することによって気相成長黒鉛繊維を得ることができる。

かようにして得られた気相成長黒鉛繊維はPAN系又はビッチ系炭素繊維から製造した黒鉛繊維と違って、年輪状のほぼ完全な黒鉛構造を持っている。すなわち本発明で使用する気相成長黒鉛繊維の繊維軸方向及び年輪の成長方向（繊維軸に直行する方向）の電気伝導性及び熱伝導性は次の通りである。

(繊維軸方向) (年輪の成長方向)

| | | |
|--|---------|-------|
| ・電気比抵抗 ($\mu\Omega \cdot \text{cm}$) | 60～100 | 10000 |
| ・熱伝導度 ($\text{kcal}/\text{m} \cdot \text{hr} \cdot ^\circ\text{C}$) | 600～900 | — |

これらの特性を有する気相成長黒鉛繊維を発煙硝酸又は濃硫酸と硝酸、塩素酸カリウム、クロム酸、過マンガン酸カリウム、過塩素酸、過酸化水素、無水硫酸又はベルオキソー硫酸アンモニウムの酸化剤との酸化性濃硫酸混合溶液で15～60°C、10～90分間処理し、得られた黒鉛層間反応生成物に多量の水を加えて水洗し、90°C以上の温度で乾燥して、次いで400～1700°Cで加熱処理することによって熱膨張気相成長黒鉛繊維を得ることが出来る。得られた熱膨張気相成長黒鉛繊維は天然黒鉛から得られた熱膨張黒鉛と同様の性質を示す。

上述の黒鉛層間反応は、濃硫酸と過酸化水素の混合溶液で処理することによって行なうことが好ましく、例えば、濃度90%以上の濃硫酸と、濃度30重量%以上の過酸化水素水を気相成長黒鉛繊維の40重量%以下、好ましくは5～20重量%となるように混合し、次いで得られた混合溶液の温度を20～50°Cに保ちながら、気相成長黒鉛繊維を徐々に加えて30分以上攪拌する方法を例示し得る。

上述のようにして得られた熱膨張性気相成長黒鉛繊維単独又は該熱膨張性気相成長黒鉛繊維と膨張性黒鉛との混合物（混合割合は気相成長黒鉛繊維50重量%以下、好ましくは10～30重量%）を400～1700°Cで加熱処理して膨張させ、次いで接着剤の存在下又は非存在下で0.1～1.0mmの厚さ及び少なくとも0.9以上、好ましくは1.0～1.5のカサ密度に、室温下で圧縮することによって可撓性黒鉛シート材料を得る。

得られた可撓性黒鉛シート材料は熱膨張気相成長黒鉛繊維がからみ合っているため、また、からみ合った熱膨

張気相成長黒鉛繊維間に熱膨張黒鉛が存在するため、厚さ方向の電気伝導性及び熱伝導性が飛跳的に高くなり、且つ優れたシート強度例えば抗張力を有する。

発明の効果

本発明の可撓性黒鉛シート材料は、シートの面方向の電気比抵抗が $700\mu\Omega\cdot\text{cm}$ 以下、好ましくは $500\mu\Omega\cdot\text{cm}$ 以下で熱伝導度が $120\text{Kcal}/\text{m}\cdot\text{hr}\cdot^\circ\text{C}$ 以上、好ましくは $150\text{Kcal}/\text{m}\cdot\text{hr}\cdot^\circ\text{C}$ 以上を示し、シートの厚さ方向の電気比抵抗が $10,000\mu\Omega\cdot\text{cm}$ 以下、好ましくは $5,000\mu\Omega\cdot\text{cm}$ 以下で熱伝導度が $10\text{Kcal}/\text{m}\cdot\text{hr}\cdot^\circ\text{C}$ 以上、好ましくは $20\text{Kcal}/\text{m}\cdot\text{hr}\cdot^\circ\text{C}$ 以上を示し、且つ抗張力が $40\text{Kg}/\text{cm}^2$ 以上、好ましくは $40\sim 70\text{Kg}/\text{cm}^2$ を有する優れた電気伝導性、熱伝導性及び強度を示す可撓性黒鉛シート材料である。

更に、本発明の可撓性黒鉛シート材料は、優れた耐熱性及び耐薬品性をも示す。

実施例-1 および 2

98%硫酸400部を冷却攪拌しながら20部の60%過酸化水素水を加え、次いで気相成長黒鉛繊維100部を $20\sim 50^\circ\text{C}$ の温度範囲内で少量づつ加えながら60分間攪拌をつづ

け層間反応を行なった。反応後、生成物は充分な量の水で洗滌を行なってから、 $100\pm 5^\circ\text{C}$ の乾燥器で乾燥を行なった。

得られた熱膨張性気相成長黒鉛繊維を単独（実施例-1）または該熱膨張性気相成長黒鉛繊維と熱膨張性黒鉛との混合物（混合割合が10:90）（実施例-2）を 1000°C の電気炉でそれぞれ加熱処理して膨張させ、次いで接着剤の非存在下で圧縮して、厚み 0.5mm 、かさ密度 1.0 の可撓性黒鉛シートを得た。そのシートの電気伝導度、熱伝導度及び抗張力は次の通りである。

表

| | 抗張力 (kg/cm^2) | 電気比抵抗 ($\mu\Omega\cdot\text{cm}$) | | 熱伝導度 ($\text{Kcal}/\text{m}\cdot\text{hr}\cdot^\circ\text{C}$) | |
|-------|------------------------------------|--|-------|---|------|
| | | 面方向 | 厚み方向 | 面方向 | 厚み方向 |
| 実施例-1 | 70 | 60 | 100 | 600 | 400 |
| 実施例-2 | 42 | 550 | 4,200 | 140 | 25 |